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CHEMICAL SYSTEMS LABORATORY TECHNICAL REPORT

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DECONTAMINATION AND DISPOSAL METHODS FOR CHEMICAL AGENTS – A LITERATURE SURVEY

by

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November 1982

US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
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A proposed explosive containment system (ECS) is to be used for demilitarization of unmarked chemical munitions in the field. Input is needed for destruction of each of the possible agents that may be encountered. Therefore, a literature survey has been made of decontamination methods for the more common agents. Based upon this survey, recommendations have been given for the best method of disposal for each agent.

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PREFACE

The work described in this report was authorized and funded by Environmental Technology Division, Demil/Disposal Branch, under the title, "Explosive Containment System. Method for Agent Identification, Decontamination and Certification, C911.01.0507." The work was begun in May 1980 and completed in September 1980.

CONTENTS

DECONTAMINATION AND DISPOSAL METHODS FOR CHEMICAL AGENTS – A LITERATURE SURVEY

I. INTRODUCTION

A request was made for a review of liquid methods applicable to disposal of certain agents by US Army Toxic and Hazardous Materials Agency (USATHAMA). This information was to be utilized in the selection of systems for decontamination/disposal in an "explosive containment system" (ECS). As presented to us, the suspected agents are contained in ordnance with a charge of explosives. The concept is to detonate the ordnance with added explosives in an ECS without down loading the agent or burster charges. The concept is primarily intended for liquid-filled rounds including gases that are liquid under pressure. Some solid-filled rounds may also be amenable for disposal by this method. Liquid and solid fills will be differentiated by X-ray technique. Specifically excluded are high explosive (HE)-filled rounds. After the explosion in the ECS, the explosion "products" may be decontaminated by either a liquid reaction or by being fed into an incinerator, with the selected option depending on logistical and engineering parameters and trade-offs. This paper supplies technical information toward evaluating the chemical requirements of a liquid decontamination option.

In the liquid decontamination option, the contents of the ECS would be decontaminated with liquid reactants to neutralize the residual intact agent(s) present. The resulting decontaminated brines could be then placed into drums and transported to an existing demilitarization facility for final disposal.

No glycolates are involved. Suspected fills include GB, GD, VX, H, HD, L, GA, HN, CG, PS, AC, CK, and BBC.

After a discussion of the general problem area, it was proposed that a complete and detailed review be accomplished, which could prove a valuable reference in other decontamination/disposal operations.

The subject of decontamination can prove to be a challenging area because of the chemical breadth of the material, the variety of situations to which it is applied, and the detailed sophistication required to draw satisfactory conclusions for reduction of chemical data to actual field or engineering practice. For example, skin decontamination, field decontamination of equipment and materiel, and decontamination for disposal operations have uniquely different criteria for selection of a satisfactory system.

The choice of decontaminants for disposal operations can be based upon some of the following requirements:

(a) High reagent capacity.

(b) Well-defined products of known toxicity.

(c) Thermal moderation (ease of control).

(d) "Reasonable" rate.

(e) Low flammability for safety in handling.

(f) Economy.

(g) Special corrosion problems.

(h) Vapor or skin toxicity.

(i) Ease of incineration.

(j) Nature of incineration products.

Actual selection of a decontaminant cannot be made until a choice is made of the relative importance of the various criteria.

Other criteria may arise in special instances. The base-line data for selection, which have involved chemical studies, include information related to (a), (b), (c), (d), (g), (i), and (j). One will infrequently encounter all of the necessary information, but certain information is usually available for systems that have been studied, allowing a preliminary judgment to be made. The following appear to be minimally necessary:

(a) Determination of heat of reaction or ad hoc demonstration of thermal control of the reaction system to be utilized (see table A-1, appendix).

(b) Determination of kinetic rate constants or ad hoc demonstration of completeness of reaction within a suitable time frame (see table A-2, appendix).

(c) Demonstration of reaction products and stoichiometry or sufficient assessment to eliminate criticality of procedure when the system is applied (see tables A-2 and A-3, appendix).

(d) Ability to calculate gravimetric factors for active ingredients at least as a worst-case assessment (see table A-2).

(e) Ability to calculate, reliably, a minimal capacity factor in volume of decontaminant per gram of agent or weight of decontaminant per gram of agent (see table A-2).

(f) Analytical procedure for residual agent.

Therefore, in respect to the special problems confronting disposal, we have attempted to assemble these critical elements for disposal systems insofar as is possible. We are providing fundamental chemistry, as well as chemistry of those systems that have been inadequately explored, to ensure that sufficiently sophisticated viewpoints become available to rationalize decontaminant choice or to be able to explore promising alternatives not yet in use. Also, we hope that the deficiencies of some of the systems can become evident by a brief description of available, but incomplete, information. The following list[1,2] provides chemical nomenclature and symbols for the agents under consideration:

(GA, Tabun) ethyl dimethylphosphoramidocyanidate

(GB, Sarin) isopropyl methylphosphonofluoridate

(GD, Soman) pinacolyl methylphosphonofluoridate

(VX) O-ethyl S-(2-diisopropylaminoethyl) methylphosphonothioate

(H, HD, mustard) 2,2'-dichloroethyl sulfide

(HN-1, nitrogen mustard) bis(2-chloroethyl)ethylamine

(HN-2) bis(2-chloroethyl)methylamine

(HN-3) tris(2-chloroethyl)amine

(L, lewisite) 2-chlorovinyldichloroarsine

(CG) phosgene

(AC) hydrocyanic acid

(CK) cyanogen chloride

(PS) chloropicrin

(BBC, CA) 2-bromobenzyl cyanide

The decontamination of each of these agents will be discussed, in turn, with specific reference to:

(a) Physical properties that are of importance in decontamination; i.e., water solubility and boiling point.

(b) Criteria for selection related to:

(1) Reaction equations.

(2) Kinetics.

(3) Heat evolution.

(4) Effectiveness.

(c) Analytical methods for determination of residual agent in decontamination solutions with respect to:

(1) Sensitivity level.

(2) Reliability.

(3) Ease of application.

In addition, a discussion will be given on whether there is a "best choice" decontaminant recommended for unknown fills or whether incineration might be the only option. Tables A-1, A-2, and A-3 also provide comparisons and summaries of some of the developed information.

2. THE DECONTAMINATION OF VARIOUS AGENTS

2.1 Ethyl dimethylphosphoramidocyanidate (GA).

2.1.1 Selected physical properties. Ethyl dimethylphosphoramidocyanidate, which is miscible with water, has a boiling point of 237°C.[3]

2.1.2 Decontamination. Destruction of the agent is most easily accomplished by hydrolysis in aqueous sodium hydroxide.

$$(H_3C)_2N-\underset{\displaystyle OC_2H_5}{\overset{\displaystyle O}{\overset{\|}{\underset{|}{P}}}}-CN + 2NaOH \longrightarrow (H_3C)_2N-\underset{\displaystyle OC_2H_5}{\overset{\displaystyle O}{\overset{\|}{\underset{|}{P}}}}-ONa + NaCN + H_2O$$

The pseudo-first-order rate constant for hydrolysis of GA has been reported as being 0.02 min^{-1} at pH 9.5 and 25°C;[4] whereas, the heat of reaction was given as -10.1 kcal/mole.[5] The hydrolysis of GA in seawater has also been studied.[6]* The hydrolysis in water proceeds by two independent paths, depending upon the pH of the water.[7] At lower values, dimethylamine is produced; whereas, at higher pH, cyanide ion is liberated. In seawater, the latter route is favored. The following data were obtained at various temperatures.

Table 1. Half Life of GA in Seawater at Three Temperatures

Temperature	$t_{½}$
°C	min
15	475
20	267
25	175

The destruction of toxic cyanide ion from hydrolyzed GA is readily accomplished by oxidation with hypochlorite in basic solution, as described below in the section on hydrocyanic acid.

* Epstein, J., Rosenblatt, D. H., Gallacio, A., and McTeague, W. F. Draft Report to Commanding General, US Army Munitions Command, ATTN: AMSMU-MS-CH, Dover, New Jersey, Subject: Chemical Disposal Operations. Summary Report on a Data Base for Predicting Consequences of Chemical Disposal Operations. 2 October 1972. UNCLASSIFIED Report.

2.1.3 Analysis.

Methods for the analysis of trace amounts of agents in decontamination solutions usually involve a preliminary extraction of the agent into an organic solvent such as chloroform or hexane. This is necessary because the large excess of salts present will often interfere in the analytical method used. No study has been reported on the recovery of trace amounts of GA from aqueous sodium hydroxide, but it is likely that the compound can be extracted by chloroform, as has been reported for the water miscible GB.[8,9] Once the GA has been extracted, there are a number of methods available for its assay. Two sensitive colorimetric procedures for the determination of organophosphates, such as GA, are the Schönemann reaction with o-dianisidine and peroxide[10] and the diisonitrosoacetone reaction[11] with sensitivities of the order of 0.2 µg/ml in the final solutions. A fluorimetric technique using indole,[12] with sensitivity comparable to that of the colorimetric techniques, has been reported. Even more sensitive are enzymatic methods[13] involving acetylcholinesterase and colorimetric, fluorimetric,[14] or electrometric measurements, but these are subject to a considerable number of interferences.[15]

Many of the interferences in the above procedures can be removed prior to analysis of the agent by gas-liquid chromatographic (GLC) or thin-layer chromatographic (TLC) methods. The GLC procedure is the one customarily used for the assay of agents in organic extracts of decontamination solutions. A variety of columns is available for this purpose.

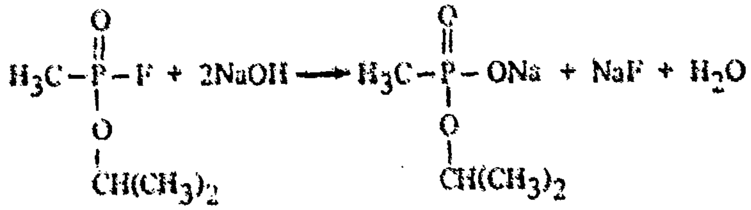
Separation prior to analysis is also possible with TLC techniques. The R_f values and semi-quantitative measurements have been made for a number of G agents using the well-known perborate-dianisidine or cholinesterase-indoxyl acetate sprays. As an alternative, the spot has been scraped off the plate and analyzed in a test tube, using standard colorimetric or fluorimetric methods.

2.2 Isopropyl methylphosphonofluoridate (GB).

2.2.1 Selected physical properties. Isopropyl methylphosphonofluoridate is completely miscible with water and boils at 151°C.[13]

2.2.2 Determination.

The most widely used method for the destruction of GB involves treatment with aqueous sodium hydroxide; i.e.,[16,17*]



The second-order reaction rate is 30 m^{-1} sec^{-1} and the heat of reaction is −44.4 kcal/mole.[16] With 5% aqueous sodium hydroxide, the half life is <0.8 sec. If the reaction mixture contains aluminum

* Kokalas, J. J., Sommer, H. Z., and Porter, G. Quarterly Progress Report. Research Plan No. 3397. Operation Red Hat. Defense Research Branch. 16 March 1973. UNCLASSIFIED Report.

metal (such as from a munition), then 10% aqueous sodium carbonate is recommended, to avoid hydrogen evolution, with a first-order rate constant of 0.08 sec^{-1}, a $t_{1/2}$ of 8.45 seconds, and a destruction efficiency of >99.9999%. The heat of the reaction (ΔH) for the carbonate process (10% sodium carbonate) has been estimated to be −22 kcal/mole.[8] This was shown to give a "safe" temperature rise of 2.58°C for an adiabatic process using 300% excess reagent (1 pound of GB per 7 gallons of 10% sodium carbonate).

$$GB + 2Na_2CO_3 + H_2O \longrightarrow CH_3-\underset{\substack{|\\ OC_3H_7}}{\overset{\substack{O\\ \|}}{P}}-ONa + NaF + 2NaHCO_3$$

Thermal tests have been conducted directly on M55 rocket warheads for the reaction of GB with sodium carbonate.* In these tests, the decontaminant was constantly flushed and agitated during the operation. Acceptable temperature rises were obtained even in the intimate presence of reacting explosives and reagents. The temperature rise was monitored by thermocouples at several locations. From an initial 74°F to 99°F, the highest temperature rise, 21°F (12°C), was at or near the decontaminant inflow. These tests were conducted under evaluation of the Johnston Island plan for disposal operations (table A-1).

Several other reviews exist which include further chemistry of GB.[18-22] The reactions are, in general, not sufficiently studied to develop full criteria for their usefulness in disposal processes. The most notable reagent that has received attention is the hypochlorite ion (present in various forms of bleach). It possesses a second-order rate coefficient for reaction with GB that is very large (10 M^{-1} sec^{-1} at 25°C). Although "catalytic" in action, much of the advantage of "catalysis" is lost by the need to use large amounts of buffer or neutralizing bases if a large capacity is to be achieved. For the hydrolytic hypochlorite reaction with GB,[18] the phosphonic acid anion and fluoride anion are produced as indicated in the previous equation. Reactions of GB with DS-2** and CD-1† (or ADP) are vigorous and rapid.[22]

The problem related to salt disposals from GB neutralization reactions with various strong bases is now well known.[23-25] The spray-drying operation results in the trace "regeneration" of GB and the need for special procedures to prevent emission of GB into the atmosphere. For this reaction, it might be desirable to produce a nonaqueous combustible product from the neutralization stage which would allow direct combustion without spray drying. This approach has not been tested. Early in the program for disposal of the war gas identification sets, the decision was made to avoid inorganic salts because of increased disposal problems caused by their presence. Consequently, monoethanolamine was widely employed as a decontaminant. However, this reagent has not been evaluated against GB, nor has the combustion of the products been studied, to assess the possibility of emission of GB reformed during that combustion process. The reaction has been investigated,[6,8] however, in connection with use of monoethanolamine as a scrubber substance.

* Kokalas, J. J., Sommer, H. Z., and Porter, G. Quarterly Progress Report. Research Plan No. 3397. Operation Red Hat. Defense Research Branch. 16 March 1973. UNCLASSIFIED Report.

** DS-2 is 2% sodium hydroxide, 28% 2-methoxyethanol, and 70% diethylenetriamine, all by weight.

† CD-1 is 2.5% lithium hydroxide hydrate, 55% 2-aminoethanol, and 45% 2-hydroxy-1-propylamine, the latter two by volume.

Decomposition rates for GB in soil and in seawater[6,26] have also been reported. The half life in seawater is about 8 hours at 25°C; whereas, for reservoir water,[27] the values at 25°C are 237 hours at pH 6.5, 75 hours at pH 7.0, 24 hours at pH 7.5, and 7.5 hours at pH 8.0. For soil, the values are 2.5 to 25 hours at 15°C depending upon moisture content.[6] Wet soil could possibly afford a moderated, useful, slow decontaminant for GB.

2.2.3 Analysis. Various reports have appeared on the analysis of GB residues in aqueous sodium hydroxide or sodium carbonate.[8,9,25,28,29] As mentioned under the analysis of GA, extraction of the brines with an organic solvent, such as chloroform, was followed by a wet chemical method of analysis[10-15] or by a GLC procedure.[8,9,25,28,29]*

2.3 Pinacolyl methylphosphonofluoridate (GD).

2.3.1 Selected physical properties. Pinacolyl methylphosphonofluoridate is appreciably less soluble in water than is GB and the boiling point is 167°C.[3]

2.3.2 Decontamination.

As with the other G agents, reaction with sodium hydroxide is considered to be the best method for use with munition fills. However, the lower aqueous solubility of GD as compared to GA and GB necessitates the use of a mixed aqueous-organic solvent if the decontamination is not to be unduly prolonged in a heterogeneous system. Because of its higher flash point, 2-methoxyethanol[30] is recommended for this purpose over methanol or ethanol and has been reported for decontamination in a mixture containing 70 parts (w/w) of the organic solvent with 30 parts (w/w) of 50% aqueous sodium hydroxide.

$$H_3C-\overset{\displaystyle O}{\overset{\|}{\underset{\underset{\displaystyle O-CH(CH_3)C(CH_3)_3}{|}}{P}}}-F + 2NaOH \longrightarrow H_3C-\overset{\displaystyle O}{\overset{\|}{\underset{\underset{\displaystyle O-CH(CH_3)C(CH_3)_3}{|}}{P}}}-ONa + NaF + H_2O$$

The reaction rate is comparable to that for GB, with a reported complete destruction within 5 minutes in excess 5% aqueous sodium hydroxide.[3] The heat of reaction should be comparable to that given for GB (−44.4 kcal/mole), as the same leaving group is involved (table A-1).

2.3.3 Analysis. Procedures for GD are similar to those described for the other organophosphates; e.g., extraction from the brine, followed by gas liquid chromatography or a colorimetric or fluorimetric analytical method.[8-15,26,28,29]

2.4 O-Ethyl S-(2-diisopropylaminoethyl) methylphosphonothioate (VX).

2.4.1 Selected physical properties. The solubility of O-ethyl S-(2-diisopropylaminoethyl) methylphosphonothioate in water is 3 gm/100 gm at 25°C and the boiling point of VX is 298°C.[13]

* Kokalas, J. J. Quarterly Progress Report. Research Plan No. 3397. Operation Red Hat. Defense Research Branch. 22 June 1973.

2.4.2 Decontamination.

Three decontamination systems have been used for bulk amounts of VX; aqueous calcium hypochlorite (HTH) slurry, acid chlorinolysis, and aqueous sodium hydroxide. Each system has its advantages and disadvantages.

The hypochlorite slurry method has the following stoichiometry:*

$$H_3C-\underset{OC_2H_5}{\overset{O}{\overset{\|}{\underset{|}{P}}}}-SCH_2CH_2N(iC_3H_7)_2 + 9OCl^- + 7OH^- \longrightarrow H_3C-\underset{OC_2H_5}{\overset{O}{\overset{\|}{\underset{|}{P}}}}-O^- +$$

$$HN(iC_3H_7)_2 + SO_4^{=} + 2CO_3^{=} + 9Cl^- + 5H_2O$$

It should be noted that the stoichiometry written for this reaction is an assumed one, which is based on partial and fragmentary evidence. The estimate for the heat of reaction also appears to have many uncertainties.** However, the calculated heat of reaction (from bond energies, etc.) of 685 kcal/mole agreed closely with an experimental value of 675 ± 13 kcal/mole.† This may be a fortuitous result. In view of some of the product complexities observed in a similar type of reaction with H (sulfur mustard, to be described later), this decontamination can only be recommended cautiously should a high degree of certainty in products and thermochemistry be required. The quoted first-order rate constant is not well defined. It is known that the reaction occurs in stepwise fashion, and the sequential stoichiometry is not known. Clearly, additional work should be performed before utilization of this reaction in a disposal process.

The experimentally determined first-order rate constant is approximately 0.01 sec^{-1}. Using the value for the heat of reaction of approximately 700 kcal/mole, the heat rise equation is $T\ (^\circ C) = \frac{\text{moles of VX } (275)}{\text{gallons of 10\% HTH}}$ (Epstein, J., 23 May 1973; see footnote). Although the reaction is theoretically a rapid one, in actuality it occurs more slowly than predicted because of the heterogeneous nature of the system. It was considered for the demilitarization of VX in a disposal project at Tooele Army Depot,[28] but it was found to be less effective than was acid chlorinolysis because of the possibility of incomplete reaction if the pH value falls below 11. To avoid this, a large excess of hypochlorite was required. Destruction of VX was then found to be better than 99.9999%.

* Epstein, J., Edgewood Arsenal. Disposition Form to Chief, Demil/Disposal Office. Heat of Reaction of VX with Alkaline Bleach Solution. 23 May 1973. UNCLASSIFIED Disposition Form.

** Pistritto, J. V. Private communication. Mr. Pistritto estimated that the calorimetric system utilized has inaccuracies of the order of 10%, even under optimal conditions, due to heat losses to the surroundings.

† Kokalas, J. J. Quarterly Progress Report. Research Plan No. 3397. Operation Red Hat. Defense Research Branch. 22 June 1973. UNCLASSIFIED Report.

A second system involves acid chlorinolysis of VX; i.e.,[31]

$$H_3C-\overset{O}{\overset{\|}{\underset{OC_2H_5}{\underset{|}{P}}}}-SCH_2CH_2N(iC_3H_7)_2 + 3Cl_2 + 4H_2O \longrightarrow H_3C-\overset{O}{\overset{\|}{\underset{OC_2H_5}{\underset{|}{P}}}}-OH + \underset{SO_3H}{\underset{|}{CH_2}}CH_2N(iC_3H_7)_2 + 6HCl$$

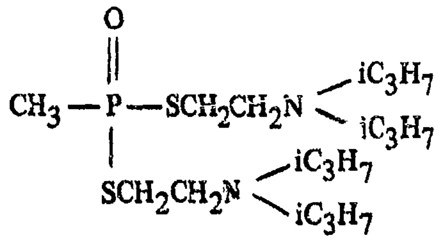
As neat VX may burn on contact with chlorine gas, the VX was first dissolved in 1.5 N hydrochloric acid. The chlorination was highly exothermic and rapid [half life ($t_{1/2}$) is 1.2 minutes at pH 4],[32] it required cooling, and it was found to be 99.999999% efficient. A major limitation of the method is the high corrosiveness of the mixture to metals, such as those that are present in the ECS.

The third decontaminant for VX is sodium hydroxide, either in an aqueous or in an organic-aqueous medium; i.e.,

$$H_3C-\overset{O}{\overset{\|}{\underset{OC_2H_5}{\underset{|}{P}}}}SCH_2CH_2N(iC_3H_7)_2 + 2NaOH \longrightarrow H_3C-\overset{O}{\overset{\|}{\underset{OC_2H_5}{\underset{|}{P}}}}-ONa + NaSCH_2CH_2N(iC_3H_7)_2 + H_2O$$

The half life of VX at pH 14 is 1.3 minutes; but, because of its low solubility in water, the reaction requires a considerably longer time unless an organic solvent such as 2-methoxyethanol is included. Using 10% aqueous hydroxide, Monsanto[33] reported the destruction of bulk amounts of VX in 6 to 8 hours at ambient temperature, with air stirring.

Similar studies were reported by the Navy.[34] A total of 12-½ gallons of VX was decontaminated using 150 gallons of 10% aqueous sodium hydroxide (air agitated) in three stages (50-gallon addition, each stage). The solubility of VX was incomplete, initially. The last two stages employed heated sodium hydroxide solutions. The time for the "complete" decontamination was 6 to 8 hours. The solubilization problem indicates that this method of decontamination will be <u>unreliable</u> unless the mixing process is very adequately controlled. It must also be noted that, if the reacting VX contains the "bis impurity"[35] (I), the action of base will generate the refractory substance (II), which will not undergo further hydrolysis.[35] This substance is highly toxic and by itself <u>would not pass Department of Transportation standards</u> for transport to a disposal site; however, the amount of bis in VX would normally be less than 10%.



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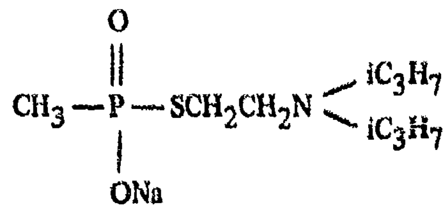

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In addition, results reported by Southern Research Insitute[22] indicate that similar caution must be applied to the toxicity of products from VX-hypochlorite reactions. In these studies, it was shown that DS-2 or CD-1 decontamination of VX yielded products with toxicities that were much lower than those achieved by the hypochlorite method. Even after 30 to 60 minutes of decontamination time, the latter mixtures remained highly toxic.

Ad hoc thermochemical (thermal profiles)* studies have been conducted for M55 warheads and M23 land mines containing VX which were allowed to react with circulated mixtures of 100 pounds of HTH in 120 gallons of water. These tests demonstrated adequate control of the temperature rise of this reaction under the specific set of conditions utilized.

The subjects of hydrolysis of VX in seawater and decomposition of VX in soil have also been reviewed.[6] In the former case, the hydrolysis of the "bis analog" (I) would give rise to the toxic refractory product (II) aforementioned. Hydrolysis in seawater would require up to 400 years to reduce VX toxicity to 1/1000 of its initial value. In soil, VX would undergo approximately 95% decomposition in 10 days with temperature, organic matter content, and perhaps moisture content being contributing factors.

Under current investigation** is the detoxification of VX using sodium dichloroisocyanurate ("Fichlor") for disposal purposes. The reaction is preferably carried out at pH 6 or lower. Products are variable, and definite kinetics cannot be established because of the sequential nature of the reactions. A precipitate of isocyanuric acid is produced as a result of reaction. Detergents are found to be ineffective in promoting solubilization of VX in the solution in order to speed up its destruction. The reaction bears similarities to the reaction of aqueous bleach with VX, but the reagent is less corrosive than bleach.

Of the three aforementioned methods, one would tentatively recommend the calcium hypochlorite slurry procedure for the ECS provided that a suitable analytical method were developed (see below). The acid chlorinolysis is too corrosive and the sodium hydroxide reaction is nonhomogeneous and relatively slow. Lack of a sufficient data base precludes consideration of sodium dichloroisocyanurate.

2.4.3 Analysis.

A variety of sensitive methods has been developed for the estimation of trace amounts of VX. However, a considerable number of problems have been encountered in their application to decontamination solutions because of the presence of numerous byproducts.† In the procedure involving acid chlorinolysis,[31] the solution was made basic to pH 10 and was extracted with dichloromethane. The VX in the extract was determined fluorimetrically using indole (sensitivity, 30 µg/l of brine), enzymatically, after TLC

* Kokalas, J. J. Quarterly Progress Report. Research Plan No. 3397. Operation Red Hat. Defense Research Branch. 22 June 1973.

** Hovanec, J. W., Davis, G. T., and Epstein, J. Chemical Systems Laboratory. Unpublished results. 1978.

† Wagner, P., Edgewood Arsenal. Demil Progress Report, Omnibus Program. 21 October 1976. UNCLASSIFIED Report.
Wagner, P., Edgewood Arsenal. Demil Progress Report, Omnibus Program. 4 February 1977. UNCLASSIFIED Report.

separation (0.5 μg/l) and by a GLC procedure (40 μg/l) with a detection limit of 4 ng. Recent laboratory experiments involving gas liquid chromatography of VX extracts from chlorinolysis brines indicated a considerable number of interferences and greatly reduced sensitivity.*

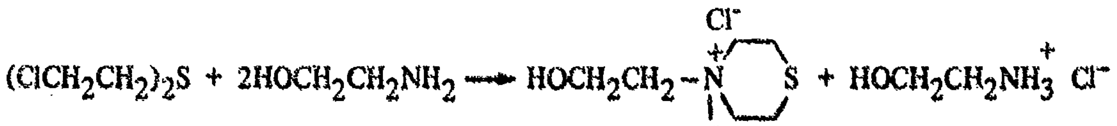
In the analysis of VX from HTH brines, problems have arisen involving poor extraction recoveries, which have not been resolved, and sensitivities have been low (600 μg/l of brine).** Yet, detailed procedures have been described for such analyses from dilute HTH solutions.[36]

2.5 2,2′-Dichloroethyl sulfide (HD).

2.5.1 Selected physical properties. The solubility of 2,2′-dichloroethyl sulfide in water is 0.1 gm/100 gm and the boiling point is 215° to 217°C.[1]

2.5.2 Decontamination.

The two most widely used decontaminants for HD are 2-aminoethanol (monoethanolamine, MEA)[37,38] and aqueous calcium hypochlorite slurry.[1] The use of MEA has a number of decided advantages† including: relatively high flash point, relatively noncorrosive to metal, inexpensive, relatively stable, homogeneous reaction with HD, moderate heat of reaction, and volume ratio of only 5:1 required. The compound is somewhat toxic (threshold limit value is 3 ppm).[39] The reaction of HD and MEA is given by the equation:



I II

The type of reaction represented by the above equation had received attention in the open literature,[40] but quantitative studies of products' kinetics and thermochemistry were not reported. A decided advantage of these systems is the absence of inorganic salts in the final disposition process.

The half life of the reaction was reported as being 11 minutes at 57°C and 40 minutes at 44°C. The heat of reaction at 50°C was -40 kcal/mole, which was the initial temperature at which the decontamination was usually carried out. Above this temperature, the heat of reaction increased significantly and cooling was required with a 5:1 v/v ratio of MEA to HD; the adiabatic temperature

* Yurow, H. W., and Valis, R. CSL unpublished results. 1979.

** Wagner, P., Edgewood Arsenal. Demil Progress Report, Omnibus Program. 21 October 1976. UNCLASSIFIED Report.
Wagner, P., Edgewood Arsenal. Demil Progress Report, Omnibus Program. 4 February 1977. UNCLASSIFIED Report.

† Crumb, E. A., Edgewood Arsenal. Disposition Form to Director of Manufacturing Technology. Decontamination of Toxic War Gas Sets. 28 March 1974. UNCLASSIFIED Disposition Form.

rises were from 50°C (initial) to 113°C (final) and 65°C (initial) to 151°C (final).* With the MEA method, residual amounts of HD from batches of 60 gallons of HD and 300 gallons of MEA were <0.25 mg of HD/l.[36]

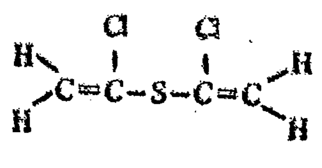
Studies have indicated that, for MEA with chloroform present (as in kit solutions and in certain munitions fills, e.g., chloropicrin), a delayed, violently exothermic reaction sometimes occurred in closed vessels,** which required that the mixture be heated in an inert atmosphere at 100°C to destroy the chloroform prior to storage. But as chloroform does not accompany HD in munitions, this treatment would not be necessary.

The above-cited hazard of a slowly appearing exotherm, which nevertheless results in a violent runaway reaction upon storage, is not an isolated instance in the history of stored materials resulting from disposal operations. Detailed methods and apparatus are now being developed for safely eliminating the appearance of such unpleasant surprises.[41] Analyses of various systems are performed by computer-controlled adiabatic calorimetry with computer data processing. Other approaches to the problem have been the previous use of DTA (differential thermal analysis) and DSC (differential scanning calorimetry), but the approach cited in the above reference develops much more complete information for analysis, if an actual problem exists. Detection of the problem should be adequately performed, however, by DTA or DSC or both. It is recommended in the present operations that such studies be run on all stored detoxified materials prior to substantial use of the method or to transportation of the resulting mixture of chemicals. Hidden exotherms in the gummy mixture from the explosive containment operation and the subsequent decontamination operation would not escape recognition.

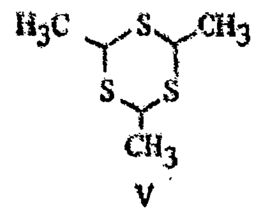
Aqueous reactions of H or HD are generally to be avoided because of the solution problems and the heterogeneous nature of the reactions. This results in uncertainty as to completeness of reactions and greater difficulties in controlling or moderating the reactions. Although bulk quantities of HD can be decontaminated with aqueous bleach (such as HTH slurry), and an assumed stoichiometry (worst case) may be utilized for the reaction, the actual products may contain many poorly identified materials whose toxicities have not been assessed.[42] For example, among the products detected from halogen reactions (usually in carbon tetrachloride) with sulfur mustard are the following:

$$ClCH{=}CH{-}S{-}CH_2CH_2Cl$$

III



IV



V

$$ClCH_2CH_2S{-}CCl{=}CHCl$$

VI

* Crumb, E. A., Edgewood Arsenal. Disposition Form to Director of Manufacturing Technology. Decontamination of Toxic War Gas Sets. 28 March 1974. UNCLASSIFIED Disposition Form.

** Crumb, E. A., Edgewood Arsenal. Disposition Form to Director of Manufacturing Technology. Decontamination of Toxic War Gas Sets. 18 December 1974. UNCLASSIFIED Disposition Form.

Cold halogenation of mustard has been shown[43] to produce an unstable adduct VII which decomposes to give a mixture of products containing VIII and IX.

$$(ClCH_2CH_2)_2-S^{\oplus}-X\ X^{\ominus}$$

VII

$$CH_2=CCl-S-CH_2CH_2Cl \qquad + \qquad ClCH=CH-S-CH_2CH_2Cl$$

VIII IX

Other reported compounds include the sulfoxide X:[44]

$$ClCH_2CH_2-\underset{\underset{O}{\|}}{S}-CHCl-CH_2Cl$$

X

Because no complete material balance on the reaction of HD mustard with various bleaches has been achieved, the absence of the above products cannot be assumed, even in aqueous solutions.

So-called "kinetics" of the reaction of bleach with mustard cannot be analytically interpreted or applied predictively on any basis other than an ad hoc situation identical to that reported. The reasons are that the stoichiometry is indefinite, and the sequential stoichiometries are unknowns. Thus, it is impossible to choose a mathematical model for the reaction. A worst-case stoichiometry can be assumed for calculation of the ultimate capacity of bleach systems. That stoichiometry is as follows:

$$(CH_2CH_2Cl)_2S + 7Ca(OCl)_2 + 2Ca(OH)_2 \longrightarrow CaSO_4 + 8CaCl_2 + 4CO_2 + 6H_2O$$

The use of aqueous sodium hydroxide solutions for decontamination of HD has been espoused;[20] but, kinetically, there is little basis for effectiveness at or near ambient temperatures. Furthermore, if hydrolyzed in water,[45] derivatives of vinyl sulfides are produced, as well as are syrupy residues containing various adducts and condensates (some of which are sulfonium derivatives) of mustard and "semi"-mustard (hydroxyethylchloroethyl sulfide), leading to a residue that can only be poorly characterized.

2.5.3 Analysis.

A technique has been reported* for the assay of HD in 2-aminoethanol. The solution was mixed with 10% aqueous sodium chloride to facilitate extraction and was extracted with hexane, which was then injected directly into a GLC system.[46] Amounts of HD down to 2.5 ppm in the analytical solution were readily determined. A similar procedure was used for the analysis of HD in

* Crumb, E. A., Edgewood Arsenal. Disposition Form to Director of Manufacturing Technology. Detection of Agents in Decontaminated Toxic War Gas Solutions. Undated. UNCLASSIFIED Disposition Form.

Crumb, E. A., Edgewood Arsenal. Disposition Form to Director of Manufacturing Technology. Simplified Methods of Analysis for H, HD, and HN-1 in Decontaminated Toxic War Gas Reaction Product Solutions. 20 March 1975. UNCLASSIFIED Disposition Form.

bleach solutions with a sensitivity of 1 ppm HD in hexane.* Further detailed procedures have been described for analysis of dilute HTH brines containing HD.[36]

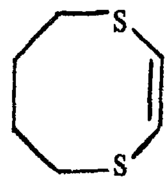
An interesting artifact substance occurring in mustard samples was found during a study of pressurizing gases for DS-2 dispersers.** This substance was not readily separated from HD by GLC and interfered with achievable sensitivity limits for analysis of the HD. Mass spectral evidence was consistent with one of the following materials:

$CH_2{=}CH-S-(CH_2)_2-S-CH{=}CH_2$

XI

$CH_3CH_2-S-CH{=}CH-S-CH{=}CH_2$

XII



XIII

This substance will therefore probably not survive the attack of oxidizing decontaminants and it poses potential interference only in the case of alkaline (base) decontaminants such as DS-2 and CD-1.

The DB-3 technique [4-(4'-nitrobenzyl)pyridine][47] has also proven to be valuable for the estimation of mustard in the hexane extract either by silica gel-impregnated glass fiber sheets or by the M18 detector kit blue-band tube. Sensitivities of HD in the MEA solution were 2.5 ppm for each of the methods.

2.6 Nitrogen mustards: bis(2-chloroethyl)ethylamine (HN-1), bis(2-chloroethyl)methylamine (HN-2), and tris(2-chloroethyl)amine (HN-3).

2.6.1 Selected physical properties. The compound HN-1 has a solubility of approximately 0.5 gm/100 gm of water at ambient temperature,[6] it has a boiling point of 85°C at 10 mm, and it decomposes on boiling at atmospheric pressure. The agent HN-2 has comparable solubility to HN-1, with a boiling point of 75°C at 10 mm. The compound HN-3 is appreciably less soluble than the other two nitrogen mustards and it boils at 138°C at 10 mm.[3]

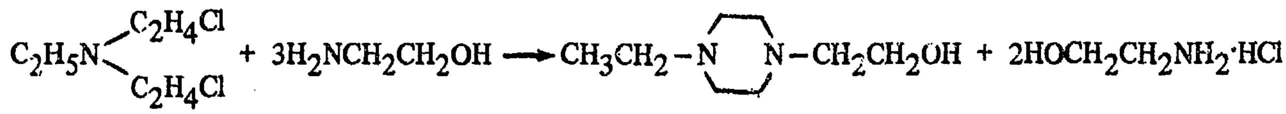
2.6.2 Decontamination.

The nitrogen mustards hydrolyze in water to give toxic products,[48] so that basic solutions are preferable for decontamination. The hydrolysis half life of HN-1 in dilute sodium hydroxide was

* Wagner, P., Edgewood Arsenal. Disposition Form. Analysis of HD in Aqueous HTH Solutions (Omnibus Program). 31 July 1973. UNCLASSIFIED Disposition Form.

** Daasch, L. W. Quarterly Progress Report. Research Plan 3350. Toxic Agent Destruction and/or Removal. Defense Research Branch, Chemical Laboratory, Edgewood Arsenal (APG). 17 September 1976. UNCLASSIFIED Report.

described as being 12 minutes at 18°C; but, because of the limited solubilities of the nitrogen mustards in this medium, a 2-aminoethanol decontaminant was preferred, with the reaction for HN-1 (and presumably for HN-2) being given as:*



The general reaction typified by the aforementioned process (amines with nitrogen mustards) has been reported in the open literature.[49,50] Kinetics have not been studied previously.

Kinetics for reaction of monoethanolamine (10 volumes of MEA in 1 volume of chloroform solution) were obtained** at 25°, 35°, and 45° with chloroform solutions containing 10% (volume) HN-1. The yield of N-ethyl-N'-hydroxyethylpiperazine (based on HN-1) was 99% ±10%.

Table 2. Reaction of HN-1 with Monoethanolamine (1 Volume of 10% HN-1 in Chloroform to 10 Volumes of Monoethanolamine)

Temperature	ko	$t_{1/2}$
°C	min^{-1}	min
25.1	0.020	34.6
34.9	0.060	11.5
44.9	0.137	5.1

The thermochemistry of this reaction has not been studied, but one might suspect similarities to the reaction of MEA and HD (table A-1, appendix).

There has been no study of aqueous oxidative methods of decontamination of the nitrogen mustards, except for some exploratory work.† A stirred chloroform solution of HN-1 in the presence of aqueous chlorine dioxide oxidant was shown to give a slow heterogeneous reaction. About 85% of the chlorine dioxide was transferred into the chloroform phase. The reaction was not further characterized.

* Defense Research Branch, Edgewood Arsenal. Disposition Form. The Decontamination of HN-1 in Chloroform in War Gas Identification Sets. 1974. UNCLASSIFIED Disposition Form.

Crumb, E. A., Edgewood Arsenal. Disposition Form to Director of Manufacturing Technology. Use of MEA as Decontaminant for Agents in Kits. 25 November 1974. UNCLASSIFIED Disposition Form.

** Pistritto, J. V., Davis, G. T., and Epstein, J. Quarterly Progress Report. Research Plan 3393. Decontamination of Agents in War Gas Identification Sets. Defense Research Branch, Chemical Laboratory, Edgewood Arsenal (APG). 10 June 1974. UNCLASSIFIED Report.

† Sarver, E. W. Quarterly Progress Report. Research Plan 3394. Defense Research Division. Decontamination of Agents in the War Gas Identification Set, Detonation, AN-M1A1, FSN 1365-323-7782. 22 June 1972. UNCLASSIFIED Report.

The reaction of nitrogen mustards in water proceeds in two steps through a substituted cyclic aziridinium* ion.[51] The first step to form the aziridinium ion is reasonably fast, but this ion is toxic[52] and relatively stable and it may be present for days or weeks in aqueous solution, depending upon the nature and concentration of nucleophiles present in solution.[51]

Values have also been obtained for the seawater solvolysis** and disappearance of HN-1. In seawater, the rate of approach to the equilibrium concentration of aziridinium ion underwent apparent increase, but the equilibrium concentration of aziridinium ion was depressed. The apparent half life of HN-1 at 4.5°C was about 25 minutes. At 25°C and pH 7.88, the half life was about 1.5 minutes in seawater. A half time of hydrolysis of all toxic materials from HN-1 was calculated to be about 12.5 days at 5°C in seawater.[6]

No study seems to have been made with HN-3 and 2-aminoethanol; but, as its general chemistry is similar to that of other nitrogen mustards, it is assumed that HN-3 can be deactivated by the reagent.

2.6.3 Analysis.

As with sulfur mustards, nitrogen mustards were determined by the DB-3 reaction. With the use of DB-3 impregnated-glass filter paper tickets,† the hexane extract of the MEA solution gave a positive test at 2.7 ppm of HN-1 in the decontamination mixture. This reagent was also employed to detect HN-1, HN-2, and HN-3 at 100 μg on TLC plates coated with silica gel.

A gas chromatographic procedure[46] (Crumb, E. A., 20 March 1975; see footnote) was developed for the assay of HN-1 in 2-aminoethanol solutions, which involved initial addition of 10% aqueous sodium chloride and extraction with hexane. Amounts of agent down to 3 ppm in 2-amino-ethanol were measured.

2.7 2-Chlorovinyldichloroarsine (lewisite, L).

2.7.1 Selected physical properties. Lewisite is relatively insoluble in water, 0.05 gm/100 gm, and it has a boiling point of 190°C (reference 1, page 290).

2.7.2 Decontamination.

Although lewisite is only slightly water soluble, it reacts rapidly and is hydrolyzed to the relatively toxic and insoluble 2-chlorovinyl arsine oxide (reference 1, page 291). More effective is aqueous sodium hydroxide, which gives the less objectionable, but still toxic, sodium arsenite. Previous disposal of arsenical products from munitions involved burial at sea.[53]

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* This ion is sometimes called the "ethyleneimmonium ion."

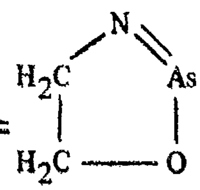
** Davis, G. T., Demek, M. M., Sarver, E. W., and Michel, H. O. Quarterly Progress Report. Research Plan 3391. Defense Research Division. Behavior of Chemical Agents Under Conditions of Chemical Disposal Operations. 22 June 1972. UNCLASSIFIED Report.

† Crumb, E. A., Edgewood Arsenal. Disposition Form to Director of Manufacturing Technology. Simplified Methods of Analysis for H, HD, and HN-1 in Decontaminated Toxic War Gas Reaction Product Solutions. 20 March 1975. UNCLASSIFIED Disposition Form.

Some reasonably comprehensive unpublished reviews of the literature on lewisite were accomplished in connection with "Decontamination of Agents in War Gas Identification Sets"* and chemical disposal.[6]** As mentioned above, hydrolysis in water is rapid to produce a gummy residue of lewisite oxide, which is in various stages of polymerization (depending upon age). Rates or mechanisms have not been studied. The thermochemistry has not been studied. The reaction with bleach is poorly characterized (Sarver, Research Plan 3394; see footnote). Stored lewisite contains three substances: lewisite I (L-1, 2-chlorovinyldichloroarsine), lewisite II (L-II, bis-2-chlorovinylchloroarsine), and arsenic trichloride. Hydrolysis in seawater is instantaneous (reference 6). Vesicant action is retained in soil for long periods of time (lewisite oxide). Oxidation of the lewisite oxide to the pentavalent state markedly reduces the toxicity. Because of the general toxicity of arsenic compounds (even in the pentavalent state), ultimate disposal presents some problems.

Reaction of lewisite with monoethanolamine has received some detailed study.† The reaction consists of a very fast process followed by a much slower reaction. The fast process has been characterized kinetically, thermochemically, and (partially) stoichiometrically.†† The slow process involves production of acetylene. The reactions are believed to be as follows:

$$ClCH{=}CH{-}AsCl_2 + H_2NCH_2CH_2OH \xrightarrow{k_1} ClCH{=}CH{-}As{=}NCH_2CH_2OH + 2HCl{\cdot}MEA \rightleftharpoons$$

$$HOCH_2CH_2N{=}As{-}NHCH_2CH_2OH + C_2H_2 + HCl{\cdot}MEA \rightleftharpoons$$  $+ MEA$

The first step has an Arrhenius activation energy (Ea) of 16.4 kcal/mole and a preexponential (A) of 3.03×10^{10} second^{-1}. The half life at 25°C is 24.4 seconds. Sensitive analyses for residual lewisite in monoethanolamine have offered some difficulties. Thermochemistry of the lewisite-MEA reaction was also studied. The heat of reaction (ΔH) was -41.0 kcal/mole based on lewisite. Products passed the Department of Transportation toxicity test for transportation (less than class B poisons).

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* Sarver, E. W. Quarterly Progress Report. Research Plan 3394. Defense Research Division. Decontamination of Agents in War Gas Identification Sets. p. 2. 10 June 1974. UNCLASSIFIED Report.

** Epstein, J., Rosenblatt, D. H., Gallacio, A., and McTeague, W. F. Draft Report to Commanding General, US Army Munitions Command, ATTN: AMSMU-MS-CH, Dover, New Jersey. Subject: Chemical Disposal Operations. Summary Report on a Data Base for Predicting Consequences of Chemical Disposal Operations. 2 October 1972. UNCLASSIFIED Report.

† Epstein, J., Eng, L., Pistritto, J. V., and Jonas, L. Status of Lewisite-Monoethanolamine Investigations. Letter Report from L. Eng, Pollutant Abatement Branch, Environmental Research Division, to Director of Manufacturing Technology, ATTN: SAREA-MT-E/Mr. J. Goheen. 24 January 1977. UNCLASSIFIED Report.

Crumb, E. A., Edgewood Arsenal. Disposition Form to Director of Manufacturing Technology. Decontamination of Dunnage from the K941-2 and K951-4 Toxic War Gas Sets. 18 March 1975. UNCLASSIFIED Disposition Form.

†† The overall stoichiometry proposed is 4 moles of MEA per mole of lewisite.

Much work has been reported on the reaction of lewisite with sodium hydroxide.* The reaction of lewisite with aqueous sodium hydroxide results in the formation of flammable acetylene; i.e.,

$$ClCH{=}CH{-}AsCl_2 + 4NaOH \longrightarrow NaAsO_2 + 3NaCl + C_2H_2\uparrow + 2H_2O$$

The decomposition was found to be essentially instantaneous (i.e., complete in <10 seconds) with a heat of reaction of -102 kcal/mole. An 18% w/v aqueous sodium hydroxide solution containing 0.1% w/v of the surfactant hexadecyltrimethylammonium chloride and 0.2% w/v of the defoamer 2-octanol was recommended for the disposal of 5% of lewisite in chloroform and the agent was assumed to be completely destroyed in 15 minutes.

Lewisite is often accompanied by appreciable amounts (ca 10%-20%) of the vesicant L II, [dichlorovinylchloroarsine $(ClCH{=}CH)_2{-}AsCl$], which seems to decompose appreciably more slowly in aqueous sodium hydroxide than does lewisite. While L II in chloroform solutions of lewisite was found to be more resistant than lewisite to aqueous sodium hydroxide, the product solution passed the standard Department of Transportation test.

2.7.3 Analysis.

The recommended assay of lewisite in aqueous sodium hydroxide was somewhat involved** and utilized the reaction of the acetylene produced from an aqueous copper (I) ammonia complex to give a red copper (I) acetylide precipitate. The precipitate was determined either iodometrically (sensitivity of 1 ppm in decontamination solution) or colorimetrically by a copper (II) ammonia complex (12 ppm).

Lewisite was also assayed by gas liquid chromatography after extraction from the aqueous phase by chloroform, but the sensitivity was quite low at 700 ppm.

2.8 Phosgene (CG).

2.8.1 Selected physical properties. The boiling point of phosgene is 8.3°C. Although the solubility of phosgene in water is relatively low, 0.03 gm/100 gm (reference 1, page 65), the compound rapidly decomposes to give water-soluble or gaseous products.

2.8.2 Decontamination.

The most useful mixture for the destruction of phosgene is aqueous sodium hydroxide† (reference 1, page 68).

* Defense Research Branch, Edgewood Arsenal. Disposition Form. Decontamination of Lewisite in Chloroform Contained in the K951-4 War Gas Identification Sets. 1974. UNCLASSIFIED Disposition Form.

** Crumb, E. A., Edgewood Arsenal. Disposition Form to Director of Manufacturing Technology. Detection of Agents in Decontaminated Toxic War Gas Solutions. Undated. UNCLASSIFIED Disposition Form.

† Crumb, E. A., Edgewood Arsenal. Disposition Form to Director of Manufacturing Technology. Decontamination of Phosgene in War Gas Identification Sets. 11 June 1974. UNCLASSIFIED Disposition Form.

The reaction of phosgene with water is also complete in less than 20 seconds. However, the low solubility of phosgene in water leads to a requirement for agitation and difficult scrubbing.

Volatility of phosgene (b.p., 8.3°C) would argue for precooling, if a safe neutralization is to be performed, whether by water or by aqueous base. Monoethanolamine was evaluated* as a decontaminant for neat phosgene and its destruction efficiency was found to be comparable to that for aqueous sodium hydroxide.

Residues from the aqueous sodium hydroxide decontamination of phosgene were shown to be nontoxic** and to pass the Department of Transportation test.

$$COCl_2 + 4NaOH \longrightarrow Na_2CO_3 + 2NaCl + 2H_2O$$

The second-order rate constant was given as 10^4 M^{-1} sec^{-1} at 25°C, which indicated that, in 10% aqueous sodium hydroxide, >99% of phosgene would be destroyed in 0.2 millisecond.† The heat of reaction was calculated to be -101 kcal/mole. Because of the low boiling point of CG, it was recommended that the reaction should be run at 0°C with several aqueous sodium hydroxide traps. One reference on the destruction of CW munitions†† reported that CG projectiles were pierced during winter under aqueous sodium and calcium hydroxide in troughs connected to a trickling tower containing the same solution.

2.8.3 Analysis.

A direct method was reported§ for the estimation of CG in aqueous sodium hydroxide, which involved reaction with a mixture of phenyl-1-naphthylamine and p-dimethylaminobenzaldehyde to give a green color at 2.5 ppm or greater.

Several GLC procedures also have been described for phosgene[54,55] with sensitivities of the order of 10^{-3} ppm in air. In addition, the DB-3 reaction[56,57] has proven to be sensitive to <0.1 ppm of phosgene in air.

2.9 Hydrocyanic acid (AC).

2.9.1 Selected physical properties. Hydrocyanic acid has a boiling point of 26°C and it is completely miscible with water.[1] The anhydrous material is subject to explosive polymerization in the presence of bases,[58] but it is relatively stable when mixed with small amounts of acids, such as phosphoric. A jet of AC is easily ignited and it burns with a blue flame.

* Crumb, E. A., Edgewood Arsenal. Disposition Form to Director of Manufacturing Technology. Decontamination of Dunnage from the K941-2 and K951-4 Toxic War Gas Sets. 18 March 1975. UNCLASSIFIED Disposition Form.

** Christensen, M. K., Physiotoxicology Branch, Toxicology Division, Biomedical Laboratory. Disposition Form to Chief, Decontamination Research Section, Defense Branch, Chemical Laboratory. Bioassay of Residues from HD/MEA and CG/NaOH Decontamination Trials. 19 February 1974. UNCLASSIFIED Disposition Form.

† Crumb, E. A., Edgewood Arsenal. Disposition Form to Director of Manufacturing Technology. Decontamination of Phosgene in War Gas Identification Sets. 11 June 1974. UNCLASSIFIED Disposition Form.

†† Sawer, E. W. Quarterly Progress Report. Research Plan 3394. Defense Research Division. Decontamination of Agents in War Gas Identification Sets. p. 2. 10 June 1974. UNCLASSIFIED Report.

§ Crumb, E. A., Edgewood Arsenal. Disposition Form to Director of Manufacturing Technology. Detection of Agents in Decontaminated Toxic War Gas Solutions. Undated. UNCLASSIFIED Disposition Form.

2.9.2 Decontamination. The method of choice for the destruction of AC involves oxidation by hypochlorite in basic solution (reference 58, page 316),[59*] according to the following equations:

$$2HCN + 5NaOCl + 4NaOH \longrightarrow N_2 + 2Na_2CO_3 + 5NaCl + 3H_2O$$

$$4HCN + 5Ca(OCl)_2 + 8NaOH \longrightarrow 2N_2 + 4CaCO_3 + 8NaCl + 6H_2O + CaCl_2$$

Other workers[60] have reported that cyanogen chloride is an intermediate in the hypochlorite attack of cyanide ion. The cyanogen chloride then hydrolyzes through isocyanate ion to ammonia (then degraded to nitrogen) and carbonate ion. The latter reaction is relatively slow. The reaction may thus be considered to consist of two steps (the first passing through CNCl):

(1) $NaCN + NaOCl \longrightarrow NaOCN + NaCl$

(2) $2NaOCN + 3NaOCl + 2HCl \longrightarrow 5NaCl + 2CO_2 + H_2O + N_2$ or

(2a) $2NaOCN + 3NaOCl + H_2O \longrightarrow 2NaHCO_3 + 3NaCl + N_2$

The second-order rate constant for the reaction was reported to be 2.67 X 10 M^{-1} sec^{-1}, the half life was given by the equation $t_{1/2}$ sec = 2.60 X 10^{-2} (OCl^-), and the heat of reaction was estimated to be -215 kcal/mole. The reaction was claimed to give >99.99% destruction of AC. Although no measurements were made of the hypochlorite solution, use of a starch-iodine indicator scrubber showed that less than 0.002% of AC or of cyanogen chloride formed from AC and hypochlorite had escaped from the system. In the laboratory setup employed (see footnote), nitrogen gas was used to slowly transfer the AC to the decontamination solution. On a larger scale, as envisioned for the ECS, problems may arise because of:

a. The high heat of reaction.

b. The low decontaminating capacity of 5% aqueous sodium hypochlorite.

c. The precipitation of calcium carbonate when HTH is used.

d. Evolution of a large volume of nitrogen gas.

e. The possibility of explosive polymerization unless the AC is initially diluted with water.

2.9.3 Analysis. Sensitive methods are available for cyanide ion[58] including a specific ion electrode[61] with a limit of 10^{-6} M. A test paper sensitive to 10 ppm of hydrogen cyanide in air and not subject to chlorine interference contains p-nitrobenzaldehyde and potassium carbonate. (See footnote.) The pyrazolone spectrophotometric method, which involves preliminary conversion of cyanide to cyanogen chloride, can measure down to 0.02 µg/ml of cyanide[62] and is widely used.

* Eng, L., Physical Research Division, Edgewood Arsenal. Disposition Form. The Demilitarization of HCN (AC). 3 May 1974. UNCLASSIFIED Disposition Form.

2.10 Cyanogen chloride (CK).

2.10.1 Selected physical properties.

The boiling point of cyanogen chloride (CK) is 13.8°C and the solubility in water is approximately 6 gm/100 gm with hydrolysis.[63]

$$CNCl + 3NaOH \longrightarrow NaCl + Na_2CO_3 + NH_3$$

The second-order rate constant for the reaction was reported as $6 \times 10^2\ M^{-1}\ min^{-1}$ and the half life* was given by the equation $t_{1/2} = 69.3\ ms/OH^-$). It was recommended that the decontamination be run at 0°C as the reaction was found to be highly exothermic. As measured with a starch-iodine bubbler mixture, with a limit of 45 µg of cyanogen chloride, destruction of agent was of the order of 99.9996%.

Other workers[64] reported the hydrolysis rate constant to be $1.55 \times 10^{-4}\ min^{-1} + 272\ (OH^-)\ min^{-1}$. Isocyanate ($OCN^-$) is implicated as an intermediate.

The experimental setup was similar to that for AC, but acid scrubbers were used to trap the ammonia evolved. The caveats mentioned for AC decontamination apply also to that for cyanogen chloride.

2.10.2 Analysis.

The standard spectrophotometric method reported for the analysis of cyanogen chloride was similar to the pyrazolone procedure for cyanide ion[62] except that no initial chlorination was required. The strongly basic decontaminant solution was neutralized with dilute hydrochloric acid and reacted with a reagent containing N-phenyl-1,3-methylpyrazolone in 4-picoline to give a blue color with a sensitivity of 5 ppm of cyanogen chloride.**

2.11 Chloropicrin (PS).

2.11.1 Selected physical properties. The solubility in water of chloropicrin[1] is 0.2 gm/100 gm and the boiling point is 112°C.

* Crumb, E. A., Edgewood Arsenal. Disposition Form to Director of Manufacturing Technology. Decontamination of Toxic War Gas Sets. 25 November 1974. UNCLASSIFIED Disposition Form.

** Crumb, E. A., Edgewood Arsenal. Disposition Form to Director of Manufacturing Technology. Detection of Agents in Decontaminated Toxic War Gas Solutions. Undated. UNCLASSIFIED Disposition Form.

2.11.2 Decontamination.

The two systems that appear to be the most useful for the decontamination of bulk amounts of chloropicrin are alcoholic sodium hydroxide[1,53] and 2-aminoethanol.* The reaction for the former is:

$$CCl_3\,NO_2 + 5NaOH \longrightarrow 3NaCl + NaNO_2 + NaHCO_3 + 2H_2O;$$

whereas, that for the latter has not been elucidated, although a pseudo-first-order rate constant of 7.47×10^{-2} min^{-1} at 34°C was obtained and a heat of reaction of -160 kcal/mole was calculated. At 50°C, the time required for destruction of >99% of chloropicrin was estimated to be less than 30 minutes.

The destruction of chloropicrin in munitions by alcoholic sodium hydroxide was rapid and was accompanied by a violent reaction that required that the decontamination vats be covered.[53] Substitution of 2-methoxyethanol with its higher boiling point for the ethanol[30] might result in a more easily controlled reaction, but the kinetics and the heat rise would have to be determined.

As mentioned previously,** one potential hazard of the 2-aminoethanol system is that a delayed violent reaction can take place in the presence of chloroform in a closed system. Therefore, for such chloropicrin fills as the CNS mixture with chloroacetophenone and chloroform, it is recommended that the chloroform be destroyed prior to storage by heating at 100°C in an inert atmosphere.

2.11.3 Analysis.

Because of the relatively low solubility of chloropicrin in water, it could be readily extracted by solvents such as chloroform from the 2-aminoethanol solution after dilution with water. Extraction efficiencies were found to be 90%. Analysis of the extract by GLC[†,65] detected down to 5 ppm of chloropicrin in the decontamination solution. The majority of colorimetric methods for chloropicrin are based upon cleavage in basic solution to give the nitrite anion, followed by a diazo coupling reaction. In one procedure,[66] the coupling involved sulfanilic acid and N-(1-naphthyl)ethylenediamine, with a detection of 3 mg/m^3 in air.

Another type of color reaction involved the condensation of chloropicrin with pyridine to give glutaconic dialdehyde,[67] with a reported sensitivity of 0.02 μg/ml.

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* Crumb, E. A., Edgewood Arsenal. Disposition Form to Director of Manufacturing Technology. Decontamination of PS in the K953-4 War Gas Identification Sets. 20 March 1975. UNCLASSIFIED Disposition Form.

Defense Research Branch, Edgewood Arsenal. Disposition Form. Decontamination of Chloropicrin (PS) in War Gas Identification Sets. 1975. UNCLASSIFIED Disposition Form.

** Crumb, E. A., Edgewood Arsenal. Disposition Form to Director of Manufacturing Techuology. Decontamination of Toxic War Gas Sets. 18 December 1974. UNCLASSIFIED Disposition Form.

† Crumb, E. A., Edgewood Arsenal. Disposition Form to Director of Manufacturing Technology. Detection of Agents in Decontaminated Toxic War Gas Solutions. Undated. UNCLASSIFIED Disposition Form.

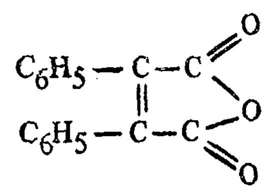
2.12 2-Bromobenzyl cyanide (BBC, formerly CA).

2.12.1 Selected physical properties. 2-Bromobenzyl cyanide is stated to be insoluble or slightly soluble in water.[1] The boiling point is 242°C with decomposition. 2-Bromobenzyl cyanide is unstable in storage except in glass.

2.12.2 Decontamination. Alcoholic sodium hydroxide[1,2,68] has been mentioned in several publications for the destruction of 2-bromobenzyl cyanide although no values for rates or for heats of reaction have been given. If the solution is partly aqueous, then the reaction is presumably:

$$C_6H_5CHBrCN + 2NaOH + H_2O \longrightarrow C_6H_5CHOHCOONa + NaBr + NH_3$$

whereas, if the alcohol is anhydrous, then one of the products formed is diphenylmaleic anhydride



It was observed* that when 2-aminoethanol and 2-bromobenzyl cyanide were mixed, a considerable amount of heat was given off, indicating that a reaction had occurred. This suggests that 2-aminoethanol is a potentially useful decontaminant for 2-bromobenzyl cyanide. However, because more is known about the alcoholic sodium hydroxide system, it remains the one of choice, with the possible substitution of 2-methoxyethanol[30] for the ethanol.

2.12.3 Analysis.

Both colorimetric and GLC methods are available for estimation of trace amounts of 2-bromobenzyl cyanide. The DB-3 method, which is a general one for alkylating agents, is capable of determining amounts of 0.25 μg/ml of the compound; whereas, the GLC technique[69] has a detection level of <3 ng, both from ethanolic solution. As 2-bromobenzyl cyanide is relatively insoluble in water, it should be possible (as it is for chloropicrin) to dilute the decontamination solution with water or with aqueous sodium chloride, followed by extraction with a solvent such as chloroform, to recover intact agent.

In addition to the above methods, it should be possible to check the efficiency of destruction of 2-bromobenzyl cyanide by a reported TLC technique employing a DB-3 spray capable of detecting 25 μg of the compound in an aqueous or an organic solvent.

2.13 Miscellaneous agents. In addition to the aforementioned liquid agents, which are the ones most likely to be present as unknowns in unmarked munitions, a number of other compounds,[1-3] although much less likely to be encountered, should be mentioned.

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* Yurow, H. W., Chemical Systems Laboratory. Unpublished results. 13 June 1980.

2.13.1 Arsenicals. There are a number of lesser known arsenic-containing agents related to lewisite.[1] They are all relatively insoluble in water (ca 0.1 gm/100 gm), but they can be readily decontaminated, as is lewisite by aqueous sodium hydroxide; e.g.,

$$CH_3AsCl_2 + 2NaOH \longrightarrow CH_3AsO + 2NaCl + H_2O$$

Included are methyl dichloroarsine (MD, CH_3AsCl_2) — boiling point, 132-133°; ethyl dichloroarsine (ED, $C_2H_5AsCl_2$) — boiling point, 156°C; phenyl dichloroarsine (PD, $C_6H_5AsCl_2$) — boiling point, 255-257°C; and diphenylchloroarsine [DA, $(C_6H_5)_2AsCl$] — boiling point, 333°C.

2.13.2 Diphosgene (DP). Also known as trichloromethyl chloroformate ($ClCOOCCl_3$), the compound is relatively insoluble in water and boils at 128°C. It is readily decomposed by aqueous sodium hydroxide, $ClCOOCl_3 + 8NaOH \longrightarrow 4NaCl + 2Na_2CO_3 + 4H_2O$. Diphosgene can be detected by all of the procedures used for phosgene.

2.13.3 Smoke agents.[2] The two most common liquid ones are chlorosulfonic acid (FS, $ClSO_3H$) and titanium tetrachloride (FM, $TiCl_4$). Both can be decontaminated by treatment with water followed by neutralization of the acid with aqueous sodium hydroxide.

2.13.4 HL mixture. For this combination munition fill (lewisite and mustard), the recommended decontaminant would be 2-aminoethanol (see reference 2).

3. BEST CHOICE DECONTAMINANT FOR UNKNOWN FILLS

Two properties of CW agents are of primary importance in their decontamination:

(a) They are almost invariably electrophilic compounds.

(b) Many of them have only slight solubility in water. Therefore, the best general reagent for their destruction should be a strong nucleophile and a good general solvent for organic compounds. One decontaminant that seems to be effective for a wide variety of agents is 2-aminoethanol,* which has been previously described in this report. Taking account of the side reaction with chloroform, it is generally superior to bleach slurries or to sodium hydroxide in water or in various solvent mixtures, which are the other two widely used multiagent decontaminants.

Incineration is also an excellent choice as a multiagent detoxification method and efficiencies have been reported for a number of agents.[70]

4. RECOMMENDED DECONTAMINANTS FOR VARIOUS AGENTS IN THE ECS SYSTEM

Based upon data described previously in this report, the following decontaminants have been recommended for the agents of interest:

(a) GA.

Aqueous sodium hydroxide is recommended unless aluminum is present; then aqueous sodium carbonate is preferred to avoid formation of hydrogen. The cyanide produced can be destroyed, if necessary,

* Crumo, E. A., Edgewood Arsenal. Disposition Form to Director of Manufacturing Technology. Use of MEA as Decontaminant for Agents in Kits. 25 November 1974. UNCLASSIFIED Disposition Form.

by subsequent treatment with aqueous calcium hypochlorite. Heat evolution should not present a problem as the ΔH is relatively low.

(b) GB.

Aqueous sodium hydroxide is recommended. The comments for GA regarding aluminum and heat evolution also apply for GB, as well as for GD below.

(c) GD.

Sodium hydroxide in a mixture of water and 2-methoxyethanol is the decontaminant of choice. The organic solvent is needed to give homogeneity.

(d) VX.

At present, no system can be recommended for ECS. However, aqueous calcium hypochlorite or Fichlor would be suitable with strong agitation if a reliable analytical method were to be developed. Fichlor is expected to react in a similar manner to that of HTH. The analysis of residual VX in dilute HTH brine is described in reference 36. Additional effort would be required to ensure that the method vigorously meets requirements.

(e) HD.

If temperature can be reasonably closely controlled in the ECS system, then 2-aminoethanol is the preferred reagent, as the reaction is homogeneous. If not, then calcium hypochlorite is suggested with the requirements of strong agitation and representative sampling.

(f) HN-1, HN-2, HN-3.

At present, no system can be recommended. However, the use of 2-aminoethanol appears to be promising, pending a study of the thermochemistry of the reaction.

(g) L.

The suggested decontaminant is 2-aminoethanol. Caution should be exercised, as acetylene is formed.

(h) CG.

Aqueous sodium hydroxide is the reagent of choice. Because of the low boiling point of phosgene, provisions for cooling may be required.

(i) AC.

Aqueous calcium hypochlorite is recommended, following an initial dilution with water, to avoid explosive polymerization.

(j) CK.

Aqueous sodium hydroxide and strong cooling are recommended for this compound.

(k) PS.

Because of insufficient data, no decontaminant can be recommended. However, the most promising appears to be sodium hydroxide in a water and 2-methoxyethanol mixture.

(l) BBC.

As with chloropicrin, insufficient data prevent a selection, but sodium hydroxide in aqueous 2-methoxyethanol shows promise.

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APPENDIX
TABLES

Table A-1. Thermochemical Data for Disposal Reactions

Agent	Decontaminant	Heat of reaction, T	Temperature rise[a] calculation already performed
		kcal/mole	
GA	Sodium hydroxide	−10.1	No
GB	Sodium hydroxide	−44.4	No
GB	10% Sodium carbonate	−22	Yes[b]
VX	Alkaline hypochlorite	Approx. −700	Yes[b,c]
VX	Acidic chlorination	Highly exothermic	No
VX	Sodium dichloroisocyanurate	Highly exothermic	No
HD (mustard)	Alkaline hypochlorite	Highly exothermic	No
HD (mustard)	Monoethanolamine (MEA)	−40 (50°C)[d]	No[e]
L (lewisite)	Monoethanolamine (MEA)	−41	No
L (lewisite)	Sodium hydroxide	−102	No
CG (phosgene)	Sodium hydroxide	−101	No
AC (hydrogen cyanide)	Alkaline bleach	−215	No
PS (chloropicrin)	Monoethanolamine	−160	No

[a] Temperature rises can be calculated for reactions with 10% HTH using a heat capacity of 1.1 cal deg^{-1} $mole^{-1}$ and assuming a certain ratio of the decontamination solution (in excess) to the agent, knowing the heat of reaction.

[b] Thermal tests on rockets also were performed.

[c] Heat rise, $\Delta T\ (^\circ C) = \dfrac{\text{moles VX} \times (275)}{\text{gallons of 10\% HTH}}$.

[d] The heat is temperature dependent and also has been determined for other temperatures than that cited in the above table.

[e] Temperature rises may be calculated for given weight ratios of reactants using heat capacities of products varying from 0.802 cal deg^{-1} gm^{-1} at 50°C to 0.823 cal deg^{-1} gm^{-1} at 86°C.

Table A-2. Rates, Gravimetric Ratios, and Capacities for Disposal Reactions

Agent	Decontaminant	Temperature	Half life	Active ingredient gravimetric ratio*	Decontaminant capacity
					gm/gm**
GA	Water (pH 9.5)	25°C	35 Minutes	–	–
GA	Seawater	25°C	175 Minutes	–	–
GB	10% Sodium carbonate	25°C	8.5 Seconds	1.51	0.066
GB	Seawater	25°C	8 Hours	–	–
GB	Soil	15°C	2.5-24 Hours	–	–
GB	Water (pH 10)	25°C	~4 Minutes	–	–
GB	5% Sodium hydroxide	25°C	<0.8 Second	0.571	0.0875
GB	DS-2	25°C	Instantaneous	Not established	
GB	5% Bleach (alkaline)	25°C	Instantaneous	~1.06	~0.047
VX	Bleach slurry (10% HTH)	25°C	~70 Seconds	~4.83	0.0207
VX	Acidic bleach (pH 4)	25°C	1.2 Minutes	0.798 (Cl_2)	Concentration dependent
VX	Water (pH 14)	25°C	1.3 Minutes	–	–
VX	Seawater	25°C	~Years	–	–
VX	Soil	–	~1 Day (95% in 10 days)	–	–
VX	DS-2	25°C	Seconds	~0.300	0.067
H, HD	Monoethanolamine (5:1)	52°C	16 Minutes	1.150	~0.2 at 5:1
HN-1	Seawater (pH 7.88)	25°C	1.5 Minutes	–	–
HN-1	Dilute sodium hydroxide	18°C	12 Minutes	–	–
HN-1	Monoethanolamine (~10% chloroform)	25°C	35 Minutes	1.876	~0.2 at 5:1
L	Water	25°C	Very fast (toxic products)	–	–
L	Seawater	25°C	Instantaneous	–	–
L	Monoethanolamine (MEA)	25°C	24 Seconds	1.176	0.2 at 5:1
L	5% Aqueous sodium hydroxide	25°C	Instantaneous	0.964	0.052 (5%)
CG (phosgene)	Water	25°C	<20 Seconds	–	–
CG (phosgene)	10% Sodium hydroxide	25°C	<<0.2 Milliseconds	1.623	0.0616
AC (hydrocyanic acid)	(10% HTH) Alkaline hypochlorite	25°C	$\frac{2.6 \times 10^{-2}}{(OCl^-)}$ Seconds	17.92	0.0056
CK (cyanogen chloride)	Aqueous sodium hydroxide	25°C	$\frac{69.2}{(OH^-)}$ Milliseconds	1.95	0.051 10% NaOH
PS (chloropicrin)	Monoethanolamine (MEA)	34°C	~10 Milliseconds	–	–
PS (chloropicrin)	10% NaOH	–	–	1.216	0.0822

* Ratio of active ingredient required to agent destroyed.

** Grams of agent destroyed per gram of full decontamination formulation.

Table A-3. Reaction Equations Used for Calculations in Table 2

$$GB + 2Na_2CO_3 + H_2O \longrightarrow NaOP(=O)(CH_3)(OCH(CH_3)_2) + NaF + 2NaHCO_3$$

$$GB + 2NaOH \longrightarrow NaOP(=O)(CH_3)(OCH(CH_3)_2) + NaF + H_2O$$

$$GB + 2NaOCl + H_2O \longrightarrow NaOP(=O)(CH_3)(OCH(CH_3)_2) + NaF + 2HCl$$

$$VX + 6Ca(OCl)_2 \cdot 4H_2O \longrightarrow CH_3-P(=O)(OC_2H_5)-O^- \ (1/2\ Ca^{++}) + HN(iC_3H_7)_2 +$$

$$CaSO_4 + 2CO_2 + 4.5CaCl_2 + 3HOCl + 24H_2O$$

$$VX + 3Cl_2 + 4H_2O \longrightarrow CH_3P(=O)(OC_2H_5)-OH + HSO_3CH_2CH_2N(iPr)_2 + 6HCl$$

$$VX + 2NaOH \longrightarrow CH_3P(=O)(OC_2H_5)-ONa + NaSCH_2CH_2N(iPr)(iPr) + H_2O$$

$$CNCl + 3NaOH \longrightarrow NaCl + Na_2CO_3 + NH_3$$

$$CCl_3NO_2 + 5NaOH \longrightarrow 3NaCl + NaNO_2 + NaHCO_3 + 2H_2O$$

$$4HCN + 9Ca(OCl)_2 \cdot 4H_2O \longrightarrow 2N_2 + 4CaCO_3 + 5CaCl_2 + 42H_2O + 8HOCl$$

Note: In some of these reactions, hypochlorite was assumed to buffer the reaction in the absence of other added bases.

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SUBJECT: Internal Request for Change in Distribution

1. This action is in response to an Edgewood Chemical Biological Center (ECBC) Internal Request for a Change in Distribution on the "Decontamination and Disposal Methods for Chemical Agents – a Literature Survey." This document was authored by Harvey W. Yurow and George T. Davis in November 1982.

2. The above listed document has a current distribution statement which limits it to US Government Agencies Only. ECBC Subject Matter Experts have reviewed the document and deem it suitable for the change in distribution to read "Approved for Public Release, Distribution is Unlimited."

3. The point of contact is Mr. Ronald L. Stafford, ECBC Security Specialist, (410) 436-6810 or ronald.l.stafford.civ@mail.mil.

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Security Manager